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Measurements of Polycyclic Aromatic Hydrocarbons in Ambient Air Particulates in Northern New England

Dartmouth Coll., Hanover, NH

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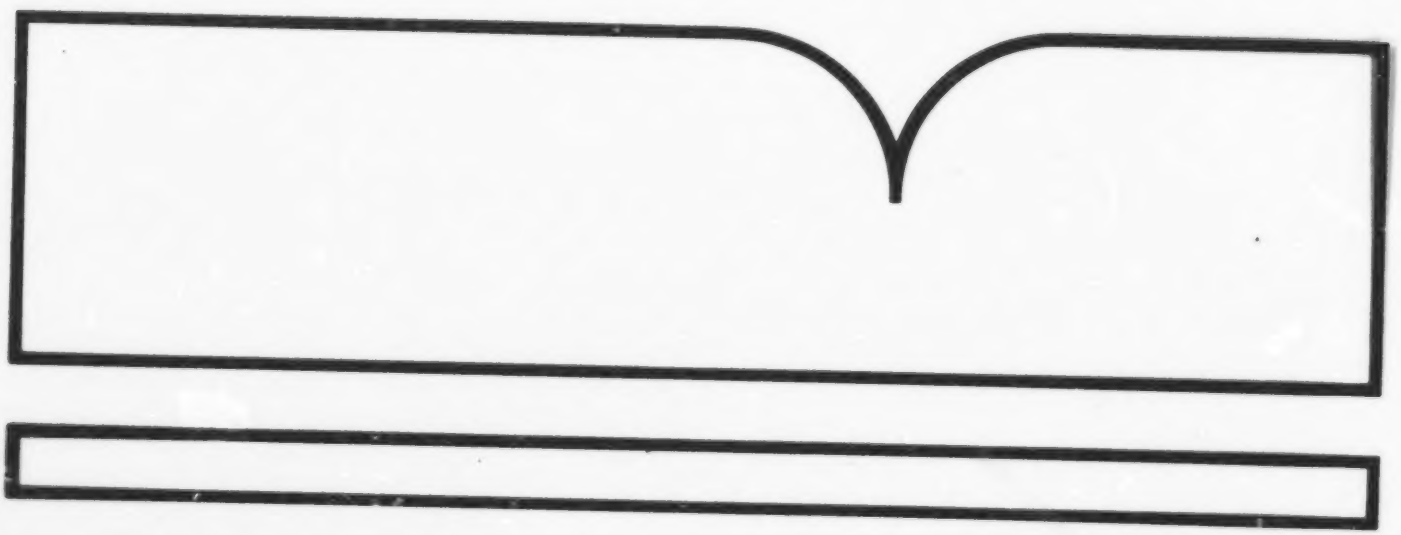

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Measurements of Polycyclic Aromatic Hydrocarbons in Ambient Air Particulates in Northern New England

by

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16. ABSTRACT The paper gives results of measuring polycyclic aromatic hydrocarbons (PAHs) in ambient air particulates in Northern New England. Standard high-volume filter samples of ambient-air particulates were collected during several heating seasons in a semi-rural village, a rural area, and in residential and institutional neighborhoods in a small twon in New Hampshire where wood is an important residential fuel. Extensive development work on extraction and cleanup schemes resulted in a reasonably convenient and reproducible procedure based on extraction of the filters with cyclohexane using ultrasonic agitation, chromatography on silica gel, and extraction of the PAH fraction into dimethylsulfoxide. Concentrations of 13 PAHs were determined by high performance liquid chromatography and by gas chromatography using both flame ionization and mass spectrometric detection. During 2 weeks of intensive sampling in late January 1982, the rural site showed the lowest PAH concentration, with Benz(a)pyrene (BaP) averaging about 1 ng/cu m, compared to about 3 ng/cu m in residential Hanover, NH. However, the variations in both the concentrations of individual PAHs among sites and the variations in the relative concentrations of the different PAHs were surprisingly small. Both the total suspended particulates and the PAH concentrations correspond well with the degree-day weather.

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a. DESCRIPTORS		b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group	
Pollution	Wood	Pollution Control	13B	11L
Aromatic Polycyclic Hydrocarbons	Fuels	Stationary Sources		21D
Measurement	Combustion	Particulate	07C	21B
Particles	Cyclohexane	New England	14B	
Aerosols	Chromatography		14G	
	Methylsulfoxide		07D	

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MEASUREMENTS OF POLYCYCLIC AROMATIC HYDROCARBONS IN AMBIENT AIR PARTICULATES IN NORTHERN NEW ENGLAND

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ABSTRACT

Standard high-volume filter samples of ambient-air particulates were collected during several heating seasons in a semi-rural village, a rural area, and in residential and institutional neighborhoods in a small town in New Hampshire where wood is an important residential fuel. Extensive development work on extraction and clean-up schemes resulted in a reasonably convenient and reproducible procedure based on extraction of the filters with cyclohexane using ultrasonic agitation, chromatography on silica gel, and extraction of the polycyclic aromatic hydrocarbon (PAH) fraction into dimethylsulfoxide. Concentrations of thirteen PAH's were determined by high performance liquid chromatography and by gas chromatography using both flame ionization and mass spectrometric detection. During a two-week period of intensive sampling in late January, 1982, the rural site showed the lowest PAH concentration, with Benz(a)pyrene (BaP) averaging about 1 ng/m^3, compared to about 3 ng/m^3 in residential Hanover, NH. However, the variation in the concentrations of individual PAH's among sites as well as variations in the relative concentrations of the different PAH's were surprisingly small. Both the total suspended particulates (TSP) and the PAH concentrations correspond well with the severity of the weather as measured by degree days.

Levoglucosan (1,6-anhydro-β-D-glucopyranose) is an important constituent of the phenolic fraction extracted from these filters and is a potentially useful qualitative and semiquantitative tracer for wood smoke; it does not arise from the burning of fossil fuels.

Introduction

The goal of this study was to measure concentrations of polycyclic aromatic hydrocarbons (PAH's) in ambient air particulates in localities which depend heavily on fuelwood. The sites chosen were a rural village without heavy traffic or industry, an area of farms and rural homes, and two sites in the larger town of Hanover, NH. The sampling was done with standard high-volume samplers; and the study included comparisons of several methods of extraction, of clean-up, and of final analysis.

Our results include measurements of absolute values of PAH concentrations at the four sites, comparisons of the relative concentrations of the different PAH's, and correlations of PAH concentrations with total suspended particulate (TSP) burdens and degree days. Finally, the results of a preliminary study of the utility of levoglucosan, a pyrolysis product of wood, as a marker compound for wood smoke are described.

Experimental Methods

Ambient-air particulates were collected on 8" x 10" Teflon-coated fiberglass filters using standard high-volume samplers (1-3). The filters were weighed to the nearest mg before and after use, and then wrapped in aluminum foil and stored at 0°F until analyzed.

The filters were extracted either with Soxhlet apparatus or with ultrasonic agitation using various solvents (cyclohexane, toluene-ethanol, benzene, methylene chloride, carbon disulfide, dimethylformamide, or dimethylsulfoxide) in order to compare the apparent extraction efficiencies and the reproducibilities of the various techniques (4-6). Ultrasonic agitation in cyclohexane was chosen for routine use. Both activated alumina and silica gel were tried for the chromatographic clean-up of the extracted samples; silica gel proved most reliable for routine use.

The final extraction and clean-up procedure is outlined in Figure 1. The filter is extracted with 200 ml of cyclohexane using ultrasonic agitation. Then the sample is concentrated by evaporation to 0.5 ml with dry N_2 gas. The remaining solution is placed on a small silica gel column, and aliphatics and PAH's are eluted together using 20 ml of cyclohexane. The PAH's are then extracted from the aliphatics with DMSO, and the layers are separated. Adding water to the DMSO layer reduces the solubility of the PAH's, and these are back-extracted into three 2 ml portions of cyclohexane. This sample is then evaporated to 0.5 ml, spiked with an internal standard, and analyzed by HPLC (Perkin Elmer Series 3/LC-GST with UV and fluorescence detection), GC/FID (Varian 3600), or GC/MS (Finnigan 4000/INCOS data system).

The recently introduced fused silica columns have revolutionized GC/MS techniques. The column is sufficiently flexible to be pushed directly through the transfer line, so that it extends continuously from the injection port to the ion source of the mass spectrometer. Quantitative delivery of the sample to the ion source is further ensured by a novel injection

technique in which the column inlet is detached and a very fine needle is used to place the sample about 5 cm into the column. For final quantitative determinations, authentic samples of all reported PAH's were used to determine individual response factors relative to an internal standard, either 2,2-binaphthyl or chrysene-d12.

The concentration of levoglucosan in the particulates was measured by extracting the filter with methanol, silating the material in the brown extract with bis(trimethylsilyl)trifluoroacetamide or bis(trimethylsilyl) acetamide. The silation products chromatograph easily, producing chromatograms having dozens or even hundreds of peaks. Triphenyl methane was used as an internal standard.

Results

The four sampling sites represent four quite different local environments. Lyme Center, New Hampshire, is a rural village with a population of 300 located in a narrow valley tributary to the Connecticut River eleven miles north of Hanover. It is on a dead-end paved road with the nearest highway with moderate traffic some three miles distant. There is no industry. A house-to-house survey in 1979-80 indicated that 60% of the homes burned substantial amounts of wood, and about 35% used wood as their primary fuel. We estimate increases to 80% and 60% respectively by 1981-82, with approximately 25 stoves within one-half mile of our sampling site. Propane is the second most common fuel, and oil a poor third.

The Brigham Hill site in Norwich, Vermont, some three miles northeast of Hanover, is in a hillside area of farms and rural homes about 500 ft. above the Connecticut River. An electrically heated house was within 100 ft. of the sampler, and another house was about 1000 ft. away. Approximately 18 stoves are located within one-half mile of the site.

There were two sampling sites in Hanover, New Hampshire (population 6000). The Ridge Road site is in a 10-square-block residential area where about 30% of the approximately 180 houses within one-half mile have stoves. The fourth site is the roof of the four-story Fairchild Science Center at Dartmouth College. Both Hanover sites are within a half-mile of two institutional power plants burning a total of 4 million gallons of No. 6 residual fuel oil per year, and both are near highways with substantial automobile and truck traffic.

During the winters of 1979-80 and 1980-81 a single high-volume sampler was rotated among several sites as we gained experience with our analytical procedures. During the winter of 1981-82, four samplers were used; and samples were collected continuously over 24-hour and 48-hour periods for the two weeks from January 16 to February 1. These 1982 measurements are the principal results of this project.

Table 1 shows the results of ultrasonic and Soxhlet extractions of identical samples with three solvents: cyclohexane, toluene/ethanol, and methylene chloride. Table 2 presents a more extensive comparison of solvents for ultrasonic extraction. None of the extractions that were tried gave complete recoveries of the spiked PAH compounds that are the focus of this study and surely are no better for recovering ambient PAH's incorporated in air particulates (6-8). Although our extractions are sufficiently reproducible so that corrections can be made for partial recoveries from spiked

filters, our results must be considered lower limits for the PAH contents of the particulates.

It should be noted that no means of collecting gaseous PAH's were employed in our sampling procedure, and that the work of others (1) as well as subsequent (1982-83) results of our own show that losses of the more volatile low molecular weight PAH's are likely to be substantial, especially on warmer days.

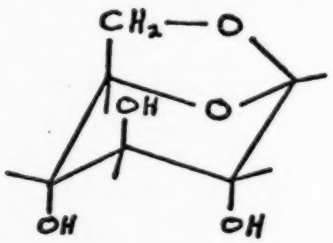
Our results show that cyclohexane, toluene/ethanol, and methylene chloride are comparable solvents for the extraction of PAH's, and that ultrasonic and Soxhlet extraction are roughly equivalent techniques. Any advantage that methylene chloride may have as a more powerful solvent is offset by greater extraction of other compounds which interefere with the PAH analysis. Our final choice of ultrasonic extraction with cyclohexane was based on overall convenience rather than on small differences in extraction efficiency.

The PAH's must be at least partially separated from the potpourri of other compounds also extracted from the particulate-laden filters before the analysis by GC or HPLC can be attempted. There are in the literature a variety of clean-up schemes using chromatography on silica gel or activated alumina (7-10), as well as a number of solvent-solvent extraction methods (11). Several of these work well in our hands, and our reasons for choosing chromatography on silica gel followed by DMSO extraction from cyclohexane solution and then addition of water and back extraction into cyclohexane as outlined in Figure 1, include convenience, reproducibility, and reasonably high yield. Overall recoveries from spiked filters are presented in Table 3.

Part of the purpose of this study was to compare gas chromatography and high pressure liquid chromatography as measurement techniques for solutions containing complex mixtures of PAH's. Resolution using fused-silica capillary-column GC was excellent, generally making the less convenient HPLC method unnecessary except when resolution of triphenylene-chrysene and of the three benzfluoranthenes was desired. Table 4 shows that both techniques are reproducible with relative standard deviations of about 5% and that they give similar quantitative results.

In contrast to the two previous winters, the winter of 1981-82 produced continuous snow cover which minimized wind-blown dust. Our two-week period of intensive sampling was ideal in that it included some of the coldest weather of the winter, averaging 63.5 degree-days per day. Comparison of degree-days, TSP values, and Benz(a)pyrene (BaP) concentrations are shown in Figure 2. Concentrations of the thirteen PAH's selected for routine analysis, averaged over the two-week sampling period, are given in Figure 3 for each of the four sites. Brigham Hill generally is the lowest with an average BaP concentration of about 3 ng/m^3. The similarity of the patterns of the relative concentrations of the thirteen PAH's was unanticipated; the Hanover sites are close to substantial amounts of both automobile and truck traffic and to domestic and institutional oil burning which was expected to produce a different mix of PAH compounds. Summertime ambient air PAH concentrations are very low (BaP usually below detection limits and always below 0.2 ng/m^3) in spite of increased vehicular traffic and reduced but still significant domestic and institutional fuel-oil consumption.

A final and interesting result was obtained during efforts to scout for the nature and approximate concentrations of polar components of ambient air particulates. A methanol extraction of the filters produced a brown extract which obviously contained phenolic and other polar material. The peak which dominates each of the complex chromatograms of the silated extracts was first thought to correspond to xylose, so-called "woodsugar". Later, by careful comparison of retention times with those of authentic samples, the peak was unequivocally associated with 1,6-anhydro-β-D-glucopyranose, commonly called levoglucosan. Levoglucosan is a well-known pyrolysis product of cellulose, wood, and starch (12).



Levoglucosan

Heating season ambient air concentrations (particulates only) are in the range 0.3-2.0 ug/m^3. Smoke samples collected from wood stoves by Professor Butcher of Bowdoin College contain large concentrations of levoglucosan (13), but those collected from coal stoves and from oil burning contain none.

Conclusions

At all four sites, the concentrations of BaP are substantially higher than those reported for rural New Hampshire and Vermont in 1970 (14). The correlations between degree-days, TSP values, and BaP concentrations (Figures 4 and 5), and the similarity of the patterns of PAH concentrations at the four sites (Figure 3) despite their rather different surroundings, together with the fact that summertime PAH levels are very low, strongly suggest to us that the dominant source of wintertime PAH's at all four sites is wood-burning.

Levoglocosan appears to hold real promise as a marker for woodsmoke. However, its quantitative usefulness will have to be demonstrated, since one might expect that the production of levoglucosan would be strongly dependent on temperature, available oxygen, and residence time during combustion. During 1982-83 we are studying the correlation between the concentrations of BaP and levoglucosan in ambient air particulates.

Acknowledgement

This work was supported in part by the Environmental Protection Agency under Grant No. R806707.

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Table 1. COMPARISON OF SOXHLET AND ULTRASONIC EXTRACTION BY THREE DIFFERENT SOLVENTS

Eighteen identical composite ambient air filter samples were used, with each extraction carried out in triplicate. Each extract was analyzed three times by GC/FID. Results are reported as the ratio to ultrasonic extraction with cyclohexane. Relative standard deviations were less than 8 percent.

Solvent	Cyclohexane		Tol/ethanol		CH_2Cl_2	
Technique*	1	2	1	2	1	2
Phenanthrene	1.00	0.81	1.06	0.79	0.77	1.15
Anthracene	1.00	0.81	1.32	1.24	0.67	1.09
Fluoranthene	1.00	0.80	1.01	0.78	0.84	1.07
Pyrene	1.00	0.84	1.00	0.80	0.82	1.14
Benz(a)anthracene	1.00	0.99	0.97	1.04	0.79	1.08
Chrysene	1.00	0.80	0.83	0.80	0.77	1.03
Benzfluoranthenes	1.00	0.87	1.05	0.94	0.79	1.06
Benz(e)pyrene	1.00	0.85	0.94	0.96	0.82	1.00
Benz(a)pyrene	1.00	0.83	0.98	0.91	0.79	1.06
Indeno(cd)pyrene	1.00	0.87	0.95	0.99	0.79	0.90
Dibenz(ah)anthracene	1.00	0.74	0.70	0.94	0.60	0.60
Benzo(ghi)perylene	1.00	0.89	1.12	1.01	0.86	1.01

*1 = Ultrasonication
2 = Soxhlet

Table 2. COMPARISON OF SOLVENTS FOR FILTER EXTRACTION

Eighteen identical composite filters were prepared, and three were used with each solvent. Results are reported relative to cyclohexane. The relative standard deviation was less than 5 percent.

C = cyclohexane, T-E = toluene/ethanol, MC = methylene chloride, B = benzene, CS_2 = carbon disulfide, DMSO = dimethyl sulfoxide

	C	T-E	MC	B	CS_2	DMSO
Phenanthrene	1.00	1.06	0.77	0.99	1.04	1.02
Anthracene	1.00	1.32	0.67	1.03	1.00	1.03
Methylphenanthrene	1.00			1.25	1.20	1.09
Fluoranthene	1.00	1.01	0.84	1.11	1.27	1.03
Pyrene	1.00	1.00	0.82	1.22	1.31	1.06
Benz(a)anthracene	1.00	0.97	0.79	1.28	1.19	0.92
Chrysene	1.00	0.83	0.77	1.18	1.27	0.97
Benzfluoranthenes	1.00	1.05	0.79	1.25	1.28	0.98
Benz(e)pyrene	1.00	0.94	0.84	1.31	1.30	1.05
Benz(a)pyrene	1.00	0.98	0.79	1.21	1.31	1.04
Indeno(cd)pyrene	1.00	0.95	0.79	1.16	1.19	1.05
Dibenz(ah)anthracene	1.00	0.70	0.60	0.70	0.73	0.63
Benzo(ghi)perylene	1.00	1.12	0.86	1.23	1.25	1.07
Coronene	1.00			1.26	0.93	1.04

Table 3. RECOVERY OF PAH USING PREFERRED PURIFICATION SCHEME

GC/MS analysis using chrysene-d 12 internal standard; 1 = recovery from simulated filter extract; 2 = recovery from spiked blank filter; 3 = recovery from spiked filter containing pre-extracted particulates

	Percent Recovery		
	1	2	3
Phenanthrene	77.4	55.5	43.0
Anthracene	62.7	50.4	55.0
Methylphenanthrene	66.9	50.8	48.7
Fluoranthene	80.7	73.7	70.2
Pyrene	79.4	70.4	67.1
Benz(a)anthracene	82.6	93.5	95.3
Chrysene	97.0	90.7	87.0
Benz(j)fluoranthene	89.7	95.3	83.9
Benz(e)pyrene	94.2	93.2	85.7
Benz(a)pyrene	91.3	97.2	85.7
Indeno(cd)pyrene	80.5	98.0	87.1
Dibenz(ah)anthracene	90.6	96.7	92.3
Benzo(ghi)perylene	83.7	91.9	80.2
Coronene	94.7	99.0	88.7

Table 4. COMPARISON OF GC AND HPLC ANALYSIS OF PAH

Average of three identical composite filters. Finnigan 4000 GC/MS; Perkin Elmer series III HPLC

	Amount found (ng) + RSD (%)*			
	GC / MS		HPLC	
Phenanthrene	580	6.2	654	10
Anthracene	44	16	62	4.8
Fluoranthene	2820	9.5	3060	5.7
Pyrene	2460	3.0	2660	5.0
Benz(a)anthracene	2730	1.8	3180	4.0
Triphenylene			1090	5.8
Chrysene			4340	4.2
Chrysene/triphenylene	5520	4.0	5430	4.2
Benz(j)fluoranthene			2560	1.3
Benz(b)fluoranthene			4550	4.6
Benz(k)fluoranthene			1870	0.75
Benzfluoranthenes	8800	4.1	8980	2.4
Benz(e)pyrene	3230	4.5	3450	3.2
Benz(a)pyrene	3030	3.1	3270	7.2
Indeno(1,2,3-cd)pyrene	3250	5.1	2830	7.9
Dibenz(ah)anthracene	480	4.8	1620	12
Benzo(ghi)perylene	3500	5.5	3560	7.4

*Relative standard deviation, N = 3.

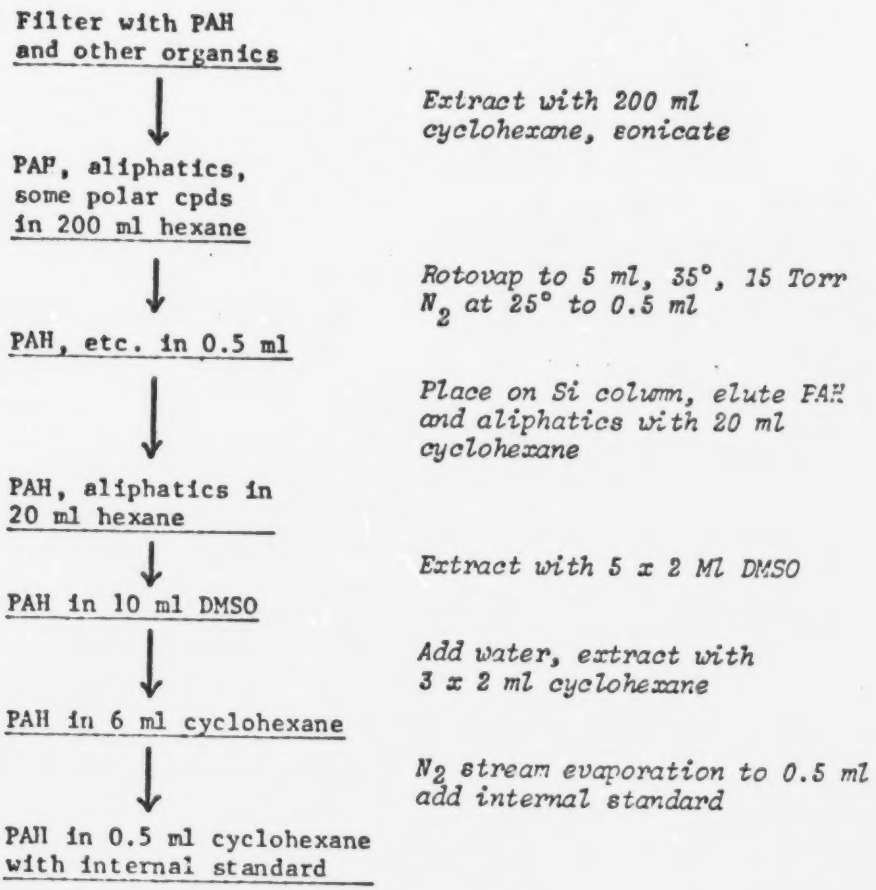
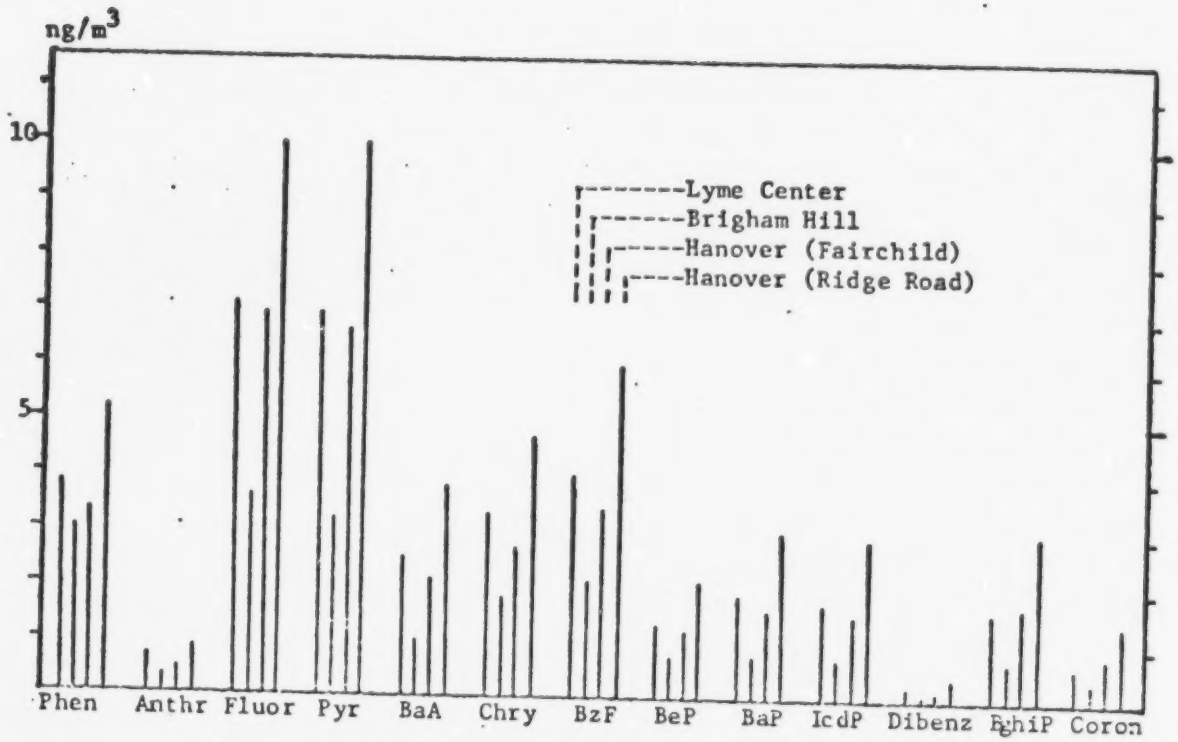

Filter with PAH and other organics
Extract with 200 ml cyclohexane, sonicate
PAH, aliphatics, some polar cpds in 200 ml hexane
Rotovap to 5 ml, 35°, 15 Torr N_2 at 25° to 0.5 ml
PAH, etc. in 0.5 ml
Place on Si column, elute PAH and aliphatics with 20 ml cyclohexane
PAH, aliphatics in 20 ml hexane
Extract with 5 x 2 Ml DMSO
PAH in 10 ml DMSO
Add water, extract with 3 x 2 ml cyclohexane
PAH in 6 ml cyclohexane
N_2 stream evaporation to 0.5 ml add internal standard
PAH in 0.5 ml cyclohexane with internal standard

Figure 1 Scheme for extracting PAH from ambient air samples.



Phen=phenanthrene, Anthr=anthracene, Fluor=fluoranthene,
Pyr=pyrene, BaA=benz(a)anthracene, Chry=chrysene,
BzF=benzfluoranthenes, BeP=benz(e)pyrene, BaP=benz(a)pyrene,
IcdP=indeno(1,2,3-cd)pyrene, Dibenz=dibenzanthracenes,
BghiP=benzo(ghi)perylene, Coron=coronene

Figure 2 Average PAH profiles: Lyme Center, Brigham Hill, Hanover (Fairchild), Hanover (Ridge Road). January 15-31, 1982.

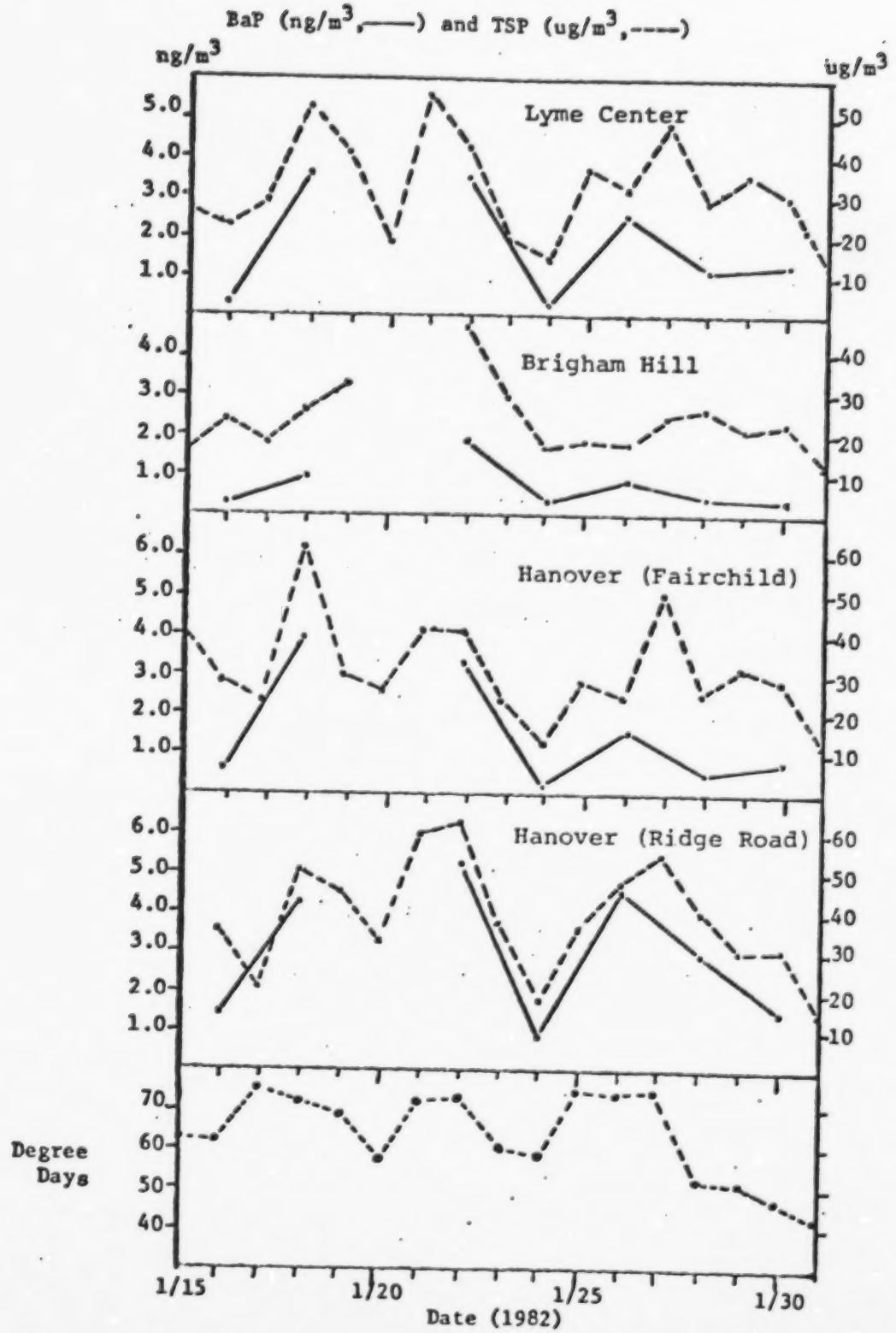


Figure 3 Benz(a)pyrene, total suspended particulates and degree days for the period January 15-31, 1982.

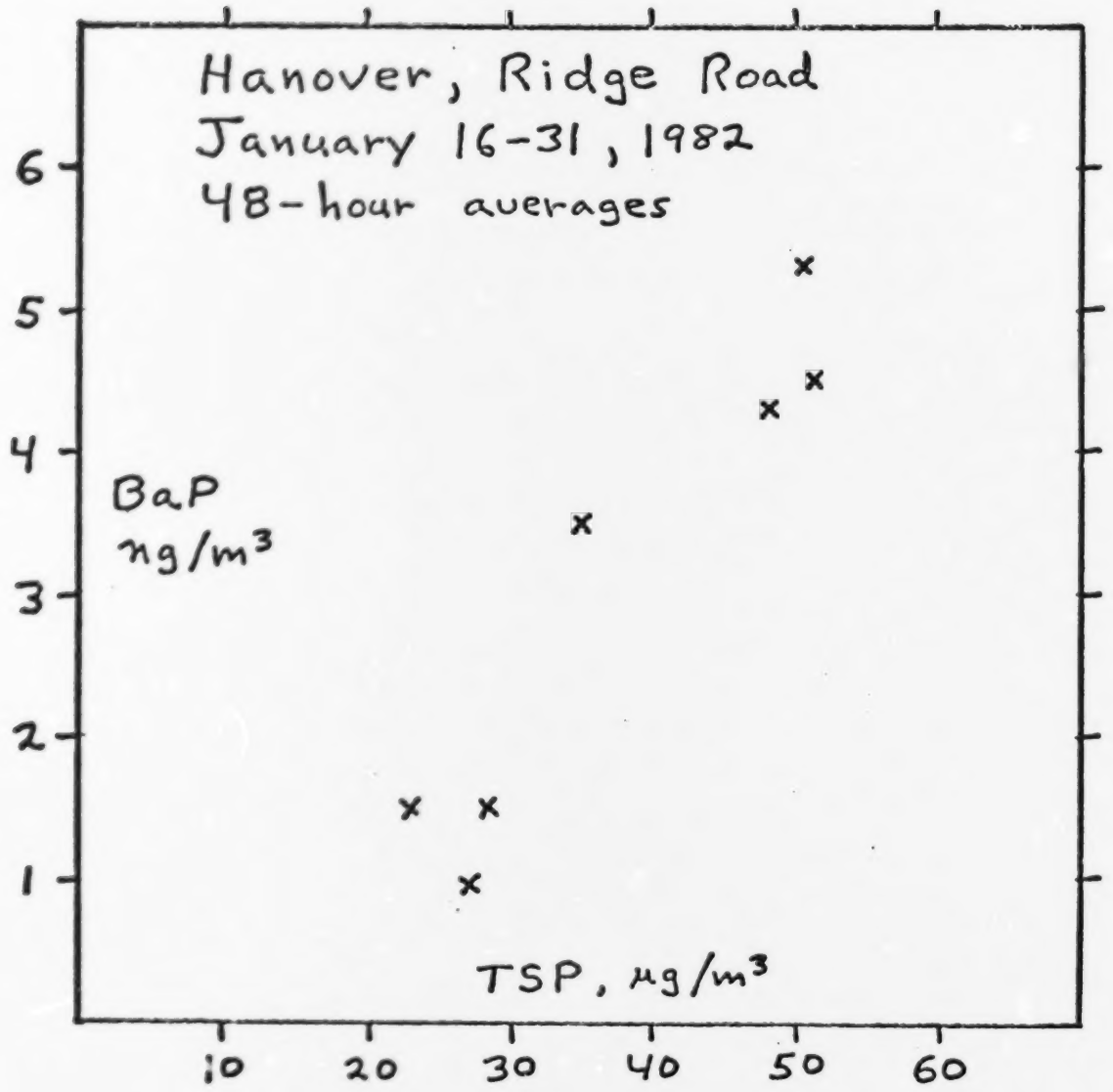


Figure 4 BaP and TSP correlation - Hanover, Ridge Road.

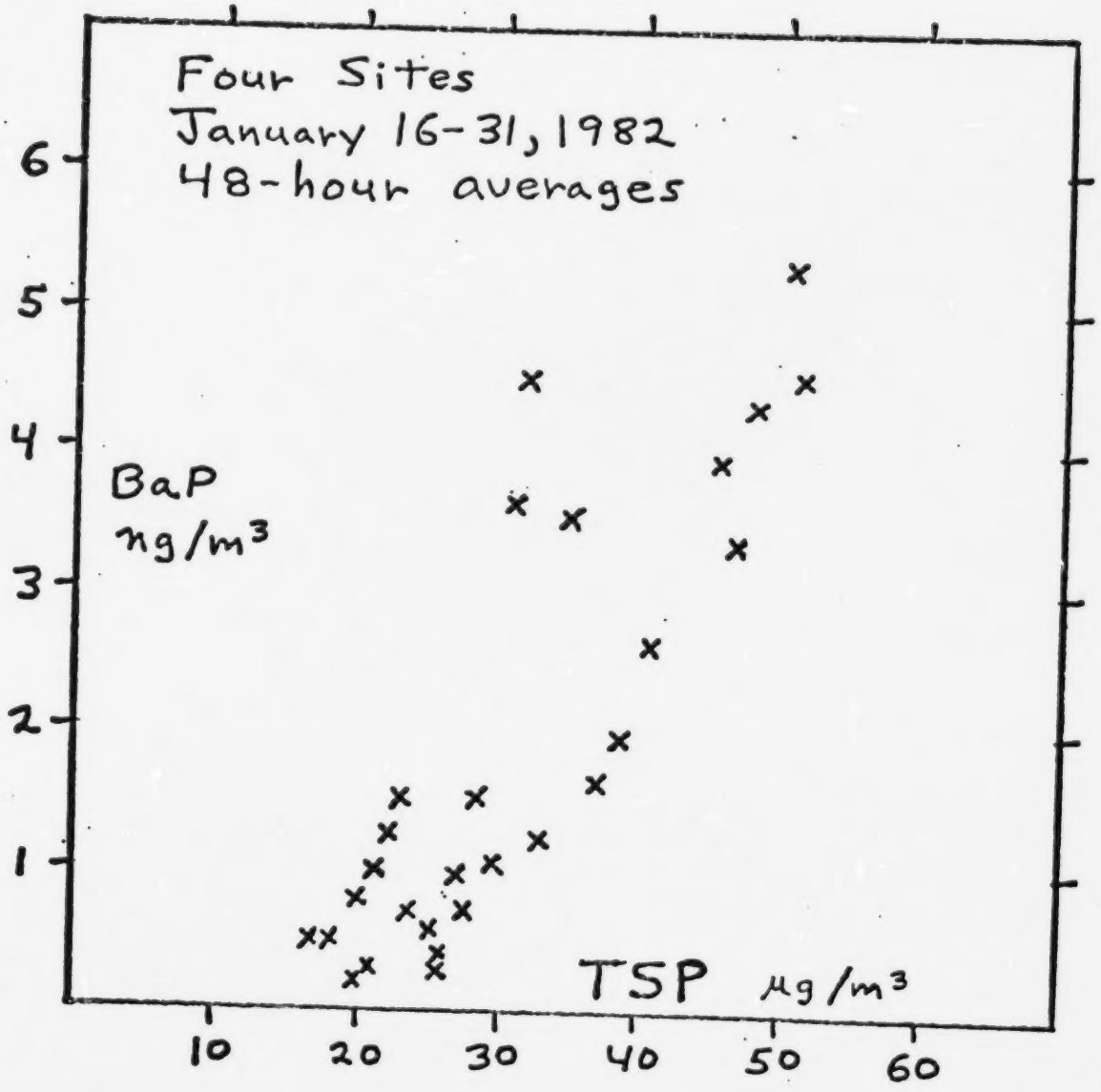


Figure 5 BaP and TSP correlation - four sites.